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(54) Title: PROCESS FOR PREPARATION OF GRANULAR DETERGENT COMPOSITION OR COMPONENT COMPRISING A WATER–SOLUBLE CATIONIC SURFACTANT

(57) Abstract

The invention provides a process which comprises the step of drying an aqueous solution or dispersion, the aqueous solution or dispersion comprising a water–soluble cationic surfactant, in the presence of a drying gas wherein the maximum temperature of the drying gas is less than 250 °C. Preferably the drying step is carried out in a co–current spray dryer and the drying gas is air.

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PROCESS FOR PREPARATION OF GRANULAR DETERGENT COMPOSITION OR COMPONENT COMPRISING A WATER-SOLUBLE CATIONIC SURFACTANT

The present invention provides a process for the preparation of granular detergent composition or component comprising a water-soluble cationic surfactant, and also provides granular detergent composition or component obtainable by the process.

Cationic surfactants are well-known detergent ingredients which are used, in particular, for imparting a soft feel to fabrics after they have been washed. The most commonly used cationic surfactants are commercially available as aqueous solutions, typically with a surfactant activity of about 35% or 40%.

Various techniques are known to dry the aqueous solutions to form powders, however these techniques have a number of disadvantages. Expensive equipment may be required, and the process may require temperatures which cause the cationic surfactant to degrade. The products prepared in this way tend to be sticky which makes subsequent handling and processing difficult. Furthermore, simply adding cationic surfactant in the form a fine powder to a granular detergent matrix significantly impairs the dispensing properties of the product for example by forming sticky, viscous gels upon contact with water.

EP-A-0 714 976, published on 5th June 1996, describes a process for spray drying cationic surfactant in the presence of low levels of anionic surfactants. However the counter-current spray drying process

described requires high temperatures which can cause some cationic surfactants to degrade.

The object of the invention is to provide a process for incorporating aqueous solutions of cationic surfactants into free-flowing, high bulk density, high active detergent granules and to avoid any degredation of the cationic surfactants, especially those with alkyl chains of C10 or less, due to high temperatures.

A further object of the invention is to provide detergent granule composition which comprise cationic surfactants and which provide good dispensing properties when used as a component of a laundry product.

Summary of the Invention

According to the invention these objects are achieved by a process which comprises the steps of :
drying an aqueous solution or dispersion, the aqueous solution or dispersion comprising a water-soluble cationic surfactant, in the presence of a drying gas wherein the maximum temperature of the drying gas is less than 250°C. Preferably the drying step is carried out in a co-current spray dryer and the drying gas is air.

Preferably the aqueous solution or dispersion also comprises water-soluble silicate. A suitable water-soluble silicate is sodium silicate having a ratio of SiO2 to Na2O is from 0.5 to 3.3, preferably from 1.0 to 2.4. Preferably the water-soluble cationic surfactant is a quaternary ammonium salt having the general formula

$$R1R2R3R4\ N^+$$

and wherein R1 is a C6 to C10 alkyl chain and R2, R3, and R4 are C1 to C7 alkyl or hydroxy alkyl chains

The invention also relates to a granular detergent composition or component comprising:

from 15% to 40%, preferably 25% to 40% by weight of water-soluble cationic surfactant;

from 5% to 85%, preferably 50% to 70% by weight of water-soluble silicate; and

from 0% to 20%, preferably 5% to 10% by weight of water.

Detailed Description of the Invention

In the process of the invention the aqueous solution or dispersion of cationic surfactant (and silicate, when present), is dried to remove water and form a free-flowing granular product. The drying step is carried out with a drying gas having a temperature of less than 250°C, preferably in a co-current spray dryer. In a spray dryer the aqueous solution or dispersion is atomised to form droplets, and the droplets are directed into a hot gas entering the drying chamber. In typical co-current spray dryers the droplets and the hot drying gas pass through the spray dryer in the same direction. Spray evaporation is rapid, the hot drying gas cools accordingly, and evaporation times are short. The cationic surfactant is not subject to heat degradation. Both rotary and nozzle-type atomizers can be used in co-current spray dryers. Most commonly the hot drying gas is air.

An alternative arrangement, is a counter-current spray dryer in which the droplets and the hot gas pass through the drying chamber in

generally opposite directions. However this arrangement often results in temperatures greater than 250°C which falls outside of the scope of the present invention.

Other means of drying the aqueous solution or dispersion of cationic surfactant may also be used provided that the maximum temperature that the cationic surfactant is exposed to does not exceed 250°C. Another such means is spray-granulation, which may be particularly advantageous because the
maximum temperature may be kept below 200°C, and preferably below 150°C.

Cationic surfactants useful in the present invention include water-soluble quaternary ammonium compounds of the form $R_1R_2R_3R_4N^+X^-$, in which R^1 is a hydroxyalkyl group having no greater than 6 carbon atoms; each of R^2 and R^3 is independently selected from C_{1-4} alkyl or alkenyl; R^4 is a C_{5-11} alkyl or alkenyl; and X^- is a counterion.

In a preferred embodiment of the invention, the cationic surfactant comprises a mixture of cationic surfactants of formula I:

$$R^1R^2\ R^3\ R^4\ N^+\ X^- \qquad (I)$$

in which R^1 is a hydroxyalkyl group having no greater than 6 carbon atoms; each of R^2 and R^3 is independently selected from C_{1-4} alkyl or alkenyl; R^4 is a C_{5-11} alkyl or alkenyl; and X^- is a counterion which does not substantially exchange with a hydroxide ion in a 0.5% by weight aqueous solution of the detergent composition at 20°C and

wherein, in the mixture of cationic surfactants of formula I , at least 10%, preferably at least 20 % or event at least 50% by weight of the cationic surfactant has R^4 which is C_{5-9} alkyl or alkenyl.

In accordance with this preferred embodiment of the invention, the cationic surfactant comprises a mixture of surfactants of formula I wherein there is a longer alkyl chain surfactant having R^4 with n carbon atoms where n is from 8 to 11 and a shorter alkyl chain surfactant having (n-2) carbon atoms.

Unless otherwise stated alkyl or alkenyl as used herein may be branched, linear or substituted. Substituents may be for example, aromatic groups, heterocyclic groups containing one or more N, S or O atoms, or halo substituents.

Silicates are well-known detergent components, and are optional components of the present invention. In particular sodium silicate is often added to detergent compositions in order to act as a corrosion inhibitor. Water-soluble silicates which are suitable for use in the present invention may be amorphous or layered.

Such silicates may be characterised by the ratio of SiO_2 to Na_2O in their structure. In the present invention, this ratio may typically be less than 3.3:1, preferably less than 2.8:1, more preferably less than 2.4:1, most preferably about 2.0:1.

In terms of the present invention, amorphous silicates are preferred to crystalline silicates. However, crystalline silicates may be included in the paste compositions of the invention.

Crystalline layered sodium silicates have the general formula

$$NaMSi_xO_{2x+1} \cdot yH_2O$$

wherein m is sodium or hydrogen, x is a number from 1.9 to 4 and y is a number from 0 to 20. Crystalline layered sodium silicates of this type are disclosed in EP-A 164 514 and methods for their preparation are disclosed in DE-A 34 17 649 and DE-A 37 42 043. For the purpose of the present invention, x in the general formula above has a value of 2, 3 or 4 and is preferably 2. More preferably M is sodium. Preferred examples of this formula comprise the α-, ß-, γ-, δ- forms of $Na_2Si_2O_5$. These materials are available from Hoechst AG, Germany, as, respectively, NaSKS-5, NaSKS-7, NaSKS-11 and NaSKS-6. The most preferred material is δ- $Na_2Si_2O_5$, NaSKS-6.

The aqueous solution or dispersion of the water-soluble cationic surfactant and the silicate, where present, in the process of the present invention may be carried out using any suitable mixing equipment. One particularly suitable piece of mixing equipment is known in the industry as a "crutcher". Other materials may be added to the crutcher at this stage, especially inorganic materials such as aluminosilicate, sulphate etc.

Finally the free-flowing granular product may be dry mixed or otherwise blended with other detergent components to form a finished detergent

composition. Typical components of a finished detergent composition include surfactants, soluble and insoluble builders, chelating agents, anti-redeposition and soil suspension agents, bleach and bleach activators, optical brighteners, soil release agents, suds suppressors, enzymes, fabric softening agents including clays, perfumes and colours, as well as other ingredients known to be useful in laundry detergents.

Examples

Granular detergent compositions were prepared as follows :

	Example 1	Example 2	Example 3	Comparative Example 4
Quaternary ammonium salt	20	28	28	20
Sodium alkyl sulphate	-	-	-	1.6
Sodium silicate (Ratio = 2.0)	28	64	-	-
Filler	44	-	65	balance
Water	8	8	7	5

In these examples the following abbreviations have been used :
Quaternary ammonium salt is C8-C10 dimethyl hydroxy ethyl ammonium chloride.
Sodium alkyl sulphate is the sodium salt of a sulphated C12-C14 linear alcohol.
Filler is sodium sulphate or sodium aluminosilicate (zeolite 4A).

The composition of Examples 1 to 3 were made by mixing aqueous solution of the quaternary ammonium salt (50% active) with an aqueous solution of the sodium silicate (45% active), and forming a homogeneous single phase mixture. The mixture was then passed through an atomising nozzle within a co-current spray-drying tower having an inlet air temperature of about 200°C. The hot granular compositions were collected at the bottom of the spray-drying tower

and allowed to cool to ambient temperature. The resulting granular compositions were found to be free-flowing.

WHAT IS CLAIMED IS:

1. A process for the preparation of a granular detergent composition or component comprising a step of drying an aqueous solution or dispersion, the aqueous solution or dispersion comprising a water-soluble cationic surfactant, wherein the drying step is carried out using a drying gas, and characterised in that the drying gas has a maximum temperature of less than 250°C.

2. A process according to claim 1 wherein the drying step is carried out in a co-current spray dryer.

3. A process according to either claim 1 or claim 2 wherein the aqueous solution or dispersion further comprises water-soluble sodium silicate, and wherein the ratio of SiO2 to Na2O is from 0.5 to 3.3.

4. A process according to claim 3 wherein the ratio of SiO2 to Na2O is from 1.0 to 2.4.

5. A process according to claim 1 wherein the water-soluble cationic surfactant is a quaternary ammonium salt having the general formula

$$R1R2R3R4\,N^{+}$$

and wherein R1 is a C6 to C10 alkyl chain and R2, R3, and R4 are C1 to C7 alkyl or hydroxy alkyl chains.

6. A granular detergent composition or component comprising:

from 15% by weight to 40% by weight of water-soluble cationic surfactant;

from 5% by weight to 85% by weight of water-soluble silicate; and

optionally, up to 20% by weight of water.

7. A granular detergent composition or component according to claim 6 wherein the water-soluble silicate is sodium silicate, and wherein the ratio of SiO2 to Na2O is from 0.5 to 3.3, preferably from 1.0 to 2.4.

8. A granular detergent composition or component according to claim 6 wherein the water-soluble cationic surfactant is a quaternary ammonium salt having the general formula

$$R1R2R3R4\ N^+$$

and wherein R1 is a C6 to C10 alkyl chain and R2, R3, and R4 are C1 to C7 alkyl or hydroxy alkyl chains.

9. A granular detergent composition or component according to any of claims 6 to 8 consisting essentially of :

from 25% by weight to 40% by weight of water-soluble cationic surfactant;

from 50% by weight to 70% by weight of water-soluble silicate; and

from 5% by weight to 10% by weight of water.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/IB98/00712

A. CLASSIFICATION OF SUBJECT MATTER

IPC(6) :C11D 11/02, 1/62, 3/08
US CL :510/443, 452, 330, 504, 511

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 510/443, 452, 330, 504, 511

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

NONE

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

APS, STN
search terms: spray drying, cationic or quaternary ammonium, silicate

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y,P	US 5,703,037 A (DOUMEN et al) 30 December 1997, abstract, col. 5, line 32 to col. 6, line 3, Example 1.	1-2
Y	US 5,259,964 A (CHAVEZ et al) 09 November 1993, abstract, col. 2, lines 57-65, col. 3, line 67 to col. 4, line 3, col. 4, lines 39-44.	1-2
Y	US 4,347,168 A (MURPHY et al) 31 August 1982, abstract, col. 2, lines 40-65, col. 4, lines 48-51, col. 4, line 61 to col. 5, line 14, col. 8, lines 62-66, col. 9, lines 19-20, col. 12, lines 10-19, examples.	1-9
Y	US 4,806,253 A (BURCKETT ST. LAURENT et al) 21 February 1989, see the entire document.	1-9

☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

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